BT-1037.11

Round Robin Cold Brittleness Tests of Balloon Films

Hauser Research & Engineering Co.

Ray L. Hauser

Sep 1966

XI. Round Robin Cold Brittleness Tests of Balloon Films

Dr. Ray L. Hauser
Hauser Research & Engineering Company
Boulder, Colorado

1. INTRODUCTION

At the 1965 AFCRL Scientific Balloon Workshop, a number of questions were raised regarding alternative methods for cold brittleness testing. A round-robin test program was suggested to compare the effects of the inclined plane and straight drop impact tests at -80°C. This paper presents the results of the round-robin program, monitored by the author and supported by the National Center for Atmospheric Research.

The inclined plane test is defined in Para. 4.5.5 of MIL-P-4640a (1965). The ball is dropped down a 60° slope which curves into a horizontal chute after a 36-in. descent. The ball then hits a vertical diaphragm of the balloon film under test.

The straight drop impact test has been developed by Winzen Research, Inc. (1964) and is included on their specification for StratoFilm, No. 320. In this case, a similar ball (2-in. diameter, 1-lb) is dropped in free-fall a distance of 27 in. to a horizontal diaphragm of test material.

In both tests, there have been questions regarding the tautness or looseness of the film in the clamps. Some experimenters have provided a slight cup to the film when it is installed at room temperature, so that thermal contraction effects can occur without putting undue stress on the film.

Interpretations of ductile or brittle failure of the films have been based largely upon the definitions given in the military specification (MIL-P-4640a, USAF, 1965). Where a definite star shatter has occurred, there has been no question of brittle failure. When the film breaks are of an intermediate geometry, the inspector's judgment of brittle or ductile failure may be the basis for acceptance.

2. MATERIALS

Film was requested from two manufacturers, and the following materials were received:

Code	Supplier	Thickness	Date or Lot	Roll
A	VisQueen X-124	0.5 mil	KF-29	----
C	VisQueen X-124	0.75 mil	KF-29	----
E	VisQueen X-124	1.0 mil	KF-29	----
G	VisQueen X-124	1.5 mil	KF-29	----
B	StratoFilm	0.5 mil	no record	----
D	StratoFilm	1.0 mil	no record	----
F	StratoFilm	1.5 mil	1343	1358

Samples of materials were coded with letter designations, and swatches 8-in. square were cut from each roll in a predetermined pattern. Ten different agencies or test methods were anticipated for this program, and five replicates of each material were prepared. The test agency/methods were numbered consecutively, and the replicate designations were made with lower case letters a, b, c, d, and e. The cutting pattern of Figure 1 was used for all films. In each case, samples a, b, and c were from the same section across the film, and samples c, d, and e were from the same section in the machine direction of the film.

Samples were mailed to each testing agency with only letter and number designations.

3. TESTING

The military specification inclined plane test is illustrated in Figure 2. This test is performed at -80°C, with a ball of diameter 2.0 ± 0.1 in. and of weight 1.0 ± 0.1 lb (a steel ball bearing 1-15/16 in. diameter meets these tolerances). The diaphragm clamp is 5 in. in diameter. Inclined plane tests were performed by VisQueen Corporation, Litton Industries, and Raven Industries.

The drop test is described by the Winzen specification (WRI Specification No. 320, 1964) and it was performed at -80°C in this program. The WRI specification describes a ball of 2-in. diameter and 1-lb weight, with the same tolerances as

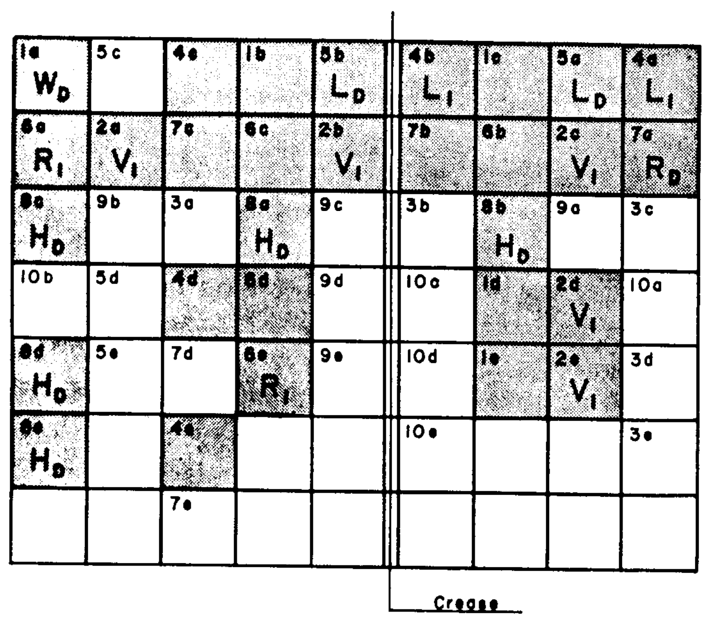

Figure 1. Pattern for Cutting Round-Robin Samples (Brittle failures, among samples tested, shaded areas - Film B, 0.5 mil StratoFilm)

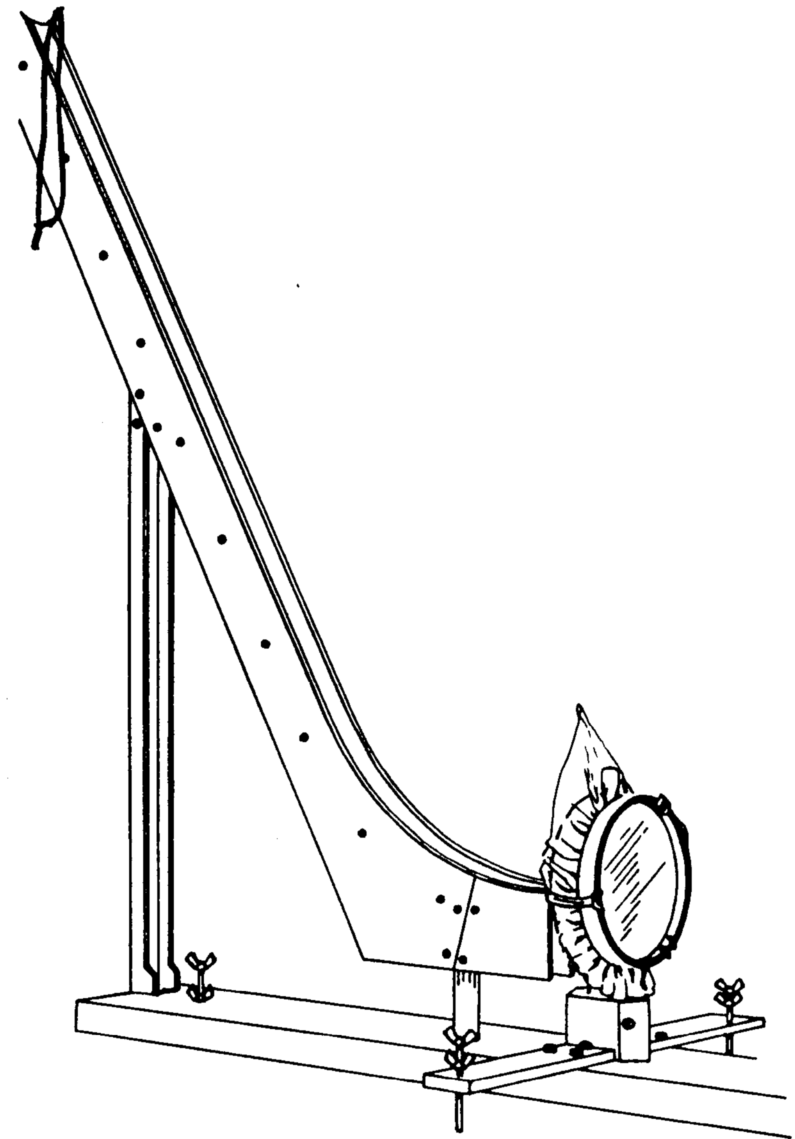

Figure 2. Inclined Plane Ball Tester

the military specification. The 27-in. free-fall was designed to provide approximately the same amount of translational impact energy to the film as the 36-in. inclined plane. Companies with facilities for the drop test were Winzen Research, Raven Industries, Litton Industries, Sea Space Systems, and Hauser Research & Engineering Company.

Companies were asked to test films in terms of their usual procedures for clamping and cupping. These conditions were reported to the monitor prior to distribution of samples. Companies were also asked to report their judgment of brittle or ductile failure, and this was done in most cases.

After testing, a number of samples were prepared for inspection of brittle or ductile failure by participants at the 1966 AFCRL Scientific Balloon Conference.

Mechanical properties of the films at -80°C were tested by the Hauser laboratory for comparison with the dynamic tests. Tensile strength and elongation were measured by methods of National Center for Atmospheric Research (1964).

4. RESULTS

The results obtained from all participants in this program are presented in Table I. Here the fraction of brittle failures is noted for each agency and test method. In some cases, less than five good tests were obtained. Drop test data from Sea Space Systems and Litton were combined into one column to provide samples of five.

Table 1. Fraction of Brittle Fractures in Round-Robin Tests at -80°C

MATERIAL	Thickness, mils	DROP TESTS 2" Ball, 27" Drop				INCL. PLANE TESTS 2" Ball, 36" Roll		
		Winzen no cup	Raven 3/8" cup	SeaSpace & Litton	Hauser no cup	Visqueen no cup	Raven 3/8" cup	Litton no cup
A, Visqueen X-124	0.55	0/5	1/5	4/5	0/5	0/5	0/5	0/5
B, StratoFilm	0.50	1/5	1/5	4/5	5/5	5/5	2/5	2/5
C, Visqueen X-124	0.75	0/3	1/5	3/4	3/5	0/5	0/5	0/5
D, StratoFilm	1.00	0/5	1/5	4/5	1/5	0/5	2/5	0/4
E, Visqueen X-124	1.00	0/4	1/5	4/6	1/5	0/5	0/5	0/5
F, StratoFilm	1.50	1/5	1/5	1/5	4/4	0/5	0/5	0/5
G, Visqueen X-124	1.50	1/5	1/5	3/5	4/4	1/5	2/5	2/5
TOTAL		3/32	7/35	23/35	18/33	6/35	6/35	4/34
		.094	.200	.657	.545	.172	.172	.118

Where results are identified only as ductile or brittle, analysis must be done in terms of fraction defective; this is not as significant, statistically, as would be a quantitative measure of brittleness.

The fraction defective (brittle) results have been treated by the standard analysis of variance techniques to learn significance of differences between test laboratories, test methods, and film variations.

The results from drop tests were quite different among the five laboratories. A comparison of Winzen and Raven results showed a difference in results at the 95 to 99 percent probability level. Even more different results were reported by Sea Space, Litton, and Hauser.

On the other hand, the ski-ball tests were very similar among the three participating laboratories. Their results were equivalent with a 99 percent probability.

The deviation coefficients for interlaboratory tests of cold brittleness were ± 40 percent for drop tests and 38 percent for inclined plane tests. These measures of precision indicate that the present cold brittleness test and inspection methods are not sound bases for acceptance/rejection criteria.

A review has been made of the number of brittle fractures in specimens from various locations in the balloon film – along the crease, in transverse directions, along the machine direction, and so forth. Among the many tests, no evidence of geometric significance was observed.

The intentional cupping of the diaphragms prior to impact gave no significant differences in test results in the inclined plane tests. Cupping and low clamping pressure may have contributed to the smaller number of brittle drop test fractures obtained by Raven and Winzen.

A map measure has been used to measure the total length of tear in each of the specimens on this program. This is suggested as a quantitative measure of brittleness to replace the subjective judgment of brittle or ductile failure. Critiques of the 17 samples which were displayed at the AFCRL conference are presented in Table 2. These samples were mostly marginal in nature, with questioned brittle or ductile failure. Some appeared to have experienced off-center impact. Persons concerned with cold brittleness testing and with specification interpretation were asked to record their judgments whether films were (a) brittle, (b) ductile, (c) questionable and another sample should be substituted, or (d) an improper test and another should be substituted. Respondents were unaware of any concern for quantitative measurement of tear length. These values are included in Table 2 for comparison with the judgments of observers.

All samples that the majority considered to be brittle failures had tear lengths exceeding 9 inches. All but one sample considered to be ductile had tear lengths less than 9 inches. One sample with 16-in. tear length had five ductile votes, three questionable votes, and one brittle vote.

Table 2. Inspection Decisions for Selected Samples From Cold Brittleness Specimens

Specimen	Inspection Decisions				Majority Opinion	Tear Length, inches
	Brittle	Ductile	Question-able, Retest	Improper Test		
B4d	II	~~IIII~~	II		Ductile	7.0
A3b	~~IIII~~ IIII				Brittle	9.0
D4a			I	~~IIII~~ III	No Test	
B4b	I	~~IIII~~ II	I		Ductile	7.4
G4e	~~IIII~~ IIII				Brittle	18.7
D4b		IIII	II	II	Ductile	6.5
A5d	I	~~IIII~~	III		Ductile	16.0
B5c	~~IIII~~ IIII				Brittle	45.7
E5c	II	~~IIII~~ II			Ductile	8.0
G6b	~~IIII~~ III	I			Brittle	19.7
G6c		~~IIII~~ IIII			Ductile	5.9
G6e	~~IIII~~ IIII				Brittle	12.6
B6a	~~IIII~~ I	II	I		Brittle	11.0
B7a	~~IIII~~ IIII				Brittle	11.5
A7b	~~IIII~~ IIII				Brittle	10.5
D6e	~~IIII~~ III				Brittle	12.5
D7b	~~IIII~~ IIII				Brittle	22.0

The tear lengths of samples A, B, and G have been measured on specimens from five laboratories. Drop test data were highly nonrandom and showed high deviation coefficients. Tear lengths from inclined plane tests were more nearly random, had lower average values and showed better precision. These data are presented in Table 3.

The criterion of a 9-in. tear length for separation of brittle or ductile behavior appears to have merit in marginal decisions.

The unidirectional mechanical properties of these films are presented in Table 4. Tests were made at -80°C with 4-in. gage length and cross-head speed 0.5 in./min. Ultimate elongations have been considered to have significance and a minimum 40 percent elongation has previously been suggested by the author as an appropriate acceptance criterion. Such a criterion would have rejected samples A and B of this program.

Table 3. Total Tear Length of Five Samples

Film	Drop Test		Incl. Plane Test	
	Length, inches	Precision, percent	Length, inches	Precision, percent
A	68.1	± 44.8	26.8	± 3.8
B	77.9	± 100	46.8	± 28.0
G	67.5	± 55.0	54.0	± 9.2

Table 4. Unidirectional Properties of Round-Robin Films at -80°C

Film	Thickness, mils	Direction	Tensile Strength, lb./in.		Elongation, percent	
			average	dev. coeff.	average	dev. coeff.
A, Visqueen X-124	0.55	machine transverse	5.51 4.99	.070 .124	88 33	.21 .46
B, StratoFilm	0.50	machine transverse	4.00 3.81	.072 .050	47 20	.33 .31
C, Visqueen X-124	0.75	machine transverse	7.27 5.99	.057 .063	150 76	.36 .78
D, StratoFilm	1.0	machine transverse	8.35 8.13	.054 .044	114 71	.31 .86
E, Visqueen X-124	1.0	machine transverse	8.67 7.87	.082 .095	106 89	.38 .68
F, StratoFilm	1.5	machine transverse	11.89 11.55	.038 .010	163 86	.42 .33
G, Visqueen X-124	1.5	machine transverse	12.26 11.92	.027 .063	177 66	.31 .63

5. CONCLUSIONS

The results of these round-robin tests indicate that the drop test is not equivalent to the ski-ball test for evaluation of cold brittleness at -80°C. The drop test is more severe as it is now being practiced by most of the laboratories (see Figure 3 and Table 5).

Cupping of the film diaphragm as much as 3/8-in. at room temperature has no significant effect on the inclined plane brittleness test at -80°C. Cupping may have some effect on the drop test.

These tests have not indicated any significant variability in cold brittleness properties of balloon films in terms of different machine or transverse locations.

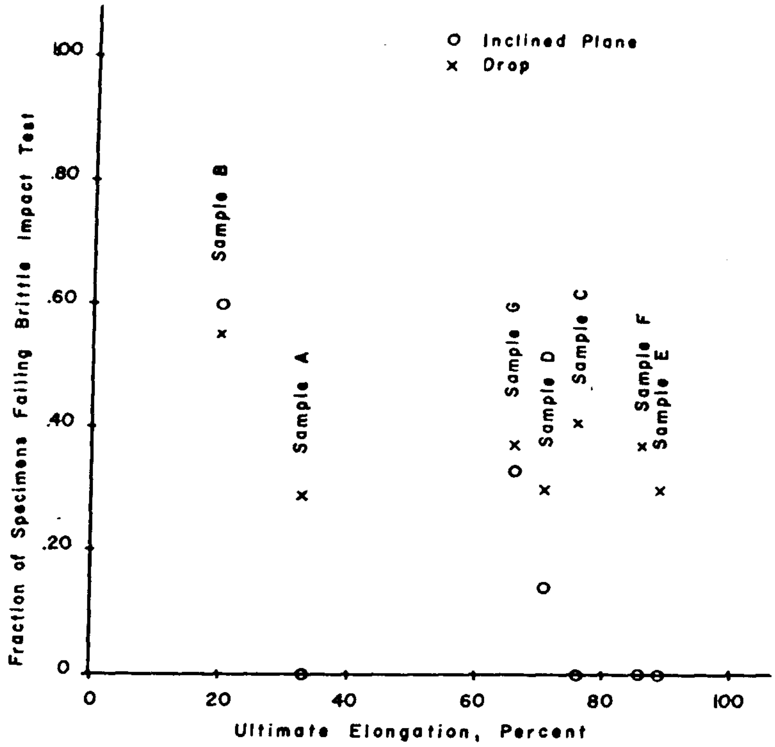

Figure 3. Correlation of Impact Brittleness and Uniaxial Elongation at -80°C

Table 5. Impact Brittleness and Elongation Tests at -80°C

Material	Thickness mils	Fraction Brittle		Ultimate Elongation, percent	
		Drop	Inclined plane	Machine direction	Transverse direction
A, Visqueen X-124	0.55	.333	.000	88	33
B, StratoFilm	0.50	.400	.600	47	20
C, Visqueen X-124	0.75	.333	.000	150	76
D, StratoFilm	1.00	.333	.143	114	71
E, Visqueen X-124	1.00	.333	.000	106	89
F, StratoFilm	1.50	.200	.000	163	86
G, Visqueen X-124	1.50	.333	.333	177	66

The average deviation coefficient for interlaboratory cold brittleness tests indicates a precision of ± 40 percent in this acceptance criterion; this is not a sound basis for acceptance to Specifications MIL-P-4640a or WRI No. 320.

A quantitative measure of cold brittleness has been proposed, and the round-robin test results indicate that a good discrimination for brittleness and ductility might be:

Tear length of four samples, total more than 36 in. – brittle,
Tear length of four samples, total less than 36 in. – ductile.